



## LABORATORY DATA CONSULTANTS, INC.

2701 Loker Ave. West, Suite 220, Carlsbad, CA 92010 Bus: 760-827-1100 Fax: 760-827-1099

Posillico Consulting  
1750 New Highway  
Farmingdale, NY 11735  
ATTN: Mr. Ellis Koch

April 24, 2014

SUBJECT: Revised Glen Isle, Data Validation

Dear Mr. Koch,

Enclosed is the revised validation report for the fraction listed below. Please replace the previously submitted report with the enclosed revised report.

**LDC Project # 31414:**

**SDG #**

480-53877-2

**Fraction**

Polychlorinated Biphenyls

- Revised report to correct the SDG number listed on page 3

Please feel free to contact us if you have any questions.

Sincerely,

Christina Rink  
Project Manager/Chemist

**Site:** Glen Isle  
**Laboratory:** Test America Buffalo, NY  
**Report No.:** 480-53877-2  
**Reviewer:** Christina Rink and Felomina Tanguilig/Laboratory Data Consultants for  
RXR Glen Isle Partners, LLC  
**Date:** April 24, 2014

**Samples Reviewed and Evaluation Summary**

FIELD ID	LAB ID	FRACTIONS VALIDATED
LT-C-028-0-2**	480-53877-1	PCBs
LT-C-028-4-6	480-53877-2	PCBs
LT-C-028-8-10	480-53877-3	PCBs
LT-C-029-0-2	480-53877-4	PCBs
LT-C-029-2-4	480-53877-5	PCBs
LT-C-029-8-10	480-53877-6	PCBs
LT-G-001-0-2	480-53877-14	PCBs
LT-G-001-4-6	480-53877-15	PCBs
LT-G-001-10-12	480-53877-16	PCBs

Associated QC Samples(s):

Field/Trip Blanks: None Associated

Field Duplicate pair: None Associated

The above-listed soil samples were collected on January 24 through January 27, 2014 and were analyzed for polychlorinated biphenyls by SW-846 method 8082A. The data validation was performed in accordance with the *USEPA Region II Functional Guidelines for Evaluating Organic Analyses* (September 2006) and the *USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review, EPA 540-R-08-01* (June 2008), modified as necessary to accommodate the non-CLP methodologies used.

The organic data were evaluated based on the following parameters:

- Data Completeness
- Holding Times and Sample Preservation
- Initial and Continuing Calibrations
- Blanks
- Surrogate Recoveries
- Matrix Spike/Matrix Spike Duplicate (MS/MSD) Results
- Laboratory Control Sample (LCS) Results
- Field Duplicate Results
- Moisture Content
- Quantitation Limits and Data Assessment
- Sample Quantitation and Compound Identification

### **Overall Evaluation of Data and Potential Usability Issues**

All results are usable as reported or usable with minor qualification due to sample matrix or laboratory quality control outliers.

Samples indicated by a double asterisk on the front cover underwent Category B review. A Category A review was performed on all of the other samples. Calibration and raw data were not evaluated for the samples reviewed by Category A criteria since this review is based on QC data.

The validation findings were based on the following information.

### **Data Completeness**

The data package was complete as defined under the requirements for the NYSDEC ASP category B laboratory deliverables.

### **Holding Times and Sample Preservation**

All criteria were met.

### **Initial and Continuing Calibrations**

Initial and continuing calibrations were not reviewed for samples reviewed by Category A criteria. Compounds that did not meet criteria in the pesticide calibrations are summarized in the following table.

Continuing calibration:

Date	Instrument ID	Column	Compound	CC %D	Associated Samples	Affected Compound		Validation Action
2/24/14	CCV (06:16)	ZB-5	Aroclor-1260	22.8	All samples in SDG 480-53877-2	Aroclor-1242 Aroclor-1248 Aroclor-1254 Aroclor-1260	XX XX XX XX	UJ nondetects UJ nondetects UJ nondetects UJ nondetects

- X = Initial calibration (IC) relative standard deviation (%RSD) > 20; estimate (J) positive and blank-qualified (UJ) results only.
- XX = Continuing calibration (CC) and second source verification percent difference (%D) > 20; estimate (J/UJ) positive and nondetect results.
- XXX = Continuing calibration (CC) and second source verification percent difference (%D) > 90; estimate (J) positive results and reject (R) nondetect results.
- = Criteria were met.

The bias cannot be determined. The results can be used for project objectives as nondetects with estimated quantitation limits (UJ) which may have a minor impact on the data usability.

### **Blanks**

Contamination was not detected in the method blanks.

A field blank was not associated with this sample set. Validation action was not required on this basis.

### **Surrogate Recoveries**

All criteria were met.

### **MS/MSD Results**

MS/MSD analyses were not performed for the PCBs analyses.

### **LCS Results**

All criteria were met.

### **Moisture Content**

All criteria were met.

### **Field Duplicate Results**

A field duplicate pair was not associated with this sample set. Validation action was not required on this basis.

**Quantitation Limits and Data Assessment**

No results were reported below the reporting limit (RL).

Dilutions were not required for PCBs analyses.

**Sample Quantitation and Compound Identification**

Calculations were spot-checked; no discrepancies were noted.

## DATA VALIDATION QUALIFIERS

- U - The analyte was analyzed for, but due to blank contamination was flagged as nondetect (U). The result is usable as a nondetect.
- J - Data are flagged (J) when a QC analysis fails outside the primary acceptance limits. The qualified “J” data are not excluded from further review or consideration. However, only one flag (J) is applied to a sample result, even though several associated QC analyses may fail. The ‘J’ data may be biased high or low or the direction of the bias may be indeterminable.
- UJ - The analyte was not detected above the reported sample quantitation limit. Data are flagged (UJ) when a QC analysis fails outside the primary acceptance limits. The qualified “UJ” data are not excluded from further review or consideration. However, only one flag is applied to a sample result, even though several associated QC analyses may fail. The ‘UJ’ data may be biased low.
- JN - The analysis indicates the presence of a compound that has been “tentatively identified” (N) and the associated numerical value represents its approximate (J) concentration.
- R - Data rejected (R) on the basis of an unacceptable QC analysis should be excluded from further review or consideration. Data are rejected when associated QC analysis results exceed the expanded control limits of the QC criteria. The rejected data are known to contain significant errors based on documented information. The data user must not use the rejected data to make environmental decisions. The presence or absence of the analyte cannot be verified.

LDC #: 31414D3b  
SDG #: 480-53877-2  
Laboratory: Test America, Inc.

# VALIDATION COMPLETENESS WORKSHEET

Cat A/Cat B

Date: 3/13/14

Page: 1 of 1

Reviewer: [Signature]

2nd Reviewer: [Signature]

**METHOD:** GC Polychlorinated Biphenyls (EPA SW 846 Method 8082) A

The samples listed below were reviewed for each of the following validation areas. Validation findings are noted in attached validation findings worksheets.

	Validation Area		Comments
I.	Technical holding times	<u>A</u>	Sampling dates: 1/24/14 - 1/27/14
II.	GC Instrument Performance Check	<u>NA</u>	Not reviewed for Cat A review.
III.	Initial calibration	<u>A</u>	Not reviewed for Cat A review. % PSD $\leq 20$
IV.	Continuing calibration/ICV	<u>SW</u>	Not reviewed for Cat A review. $CV \leq 20$
V.	Blanks	<u>A</u>	
VI.	Surrogate spikes	<u>A</u>	
VII.	Matrix spike/Matrix spike duplicates	<u>N</u>	client specified
VIII.	Laboratory control samples	<u>A</u>	LC5
IX.	Regional quality assurance and quality control	<u>N</u>	
X.	Florisil cartridge check	<u>N</u>	
XI.	GPC Calibration	<u>N</u>	
XII.	Target compound identification	<u>A</u>	Not reviewed for Cat A review.
XIII.	Compound quantitation/RL/LOQ/LODs	<u>A</u>	Not reviewed for Cat A review. no result < RL
XIV.	Overall assessment of data	<u>A</u>	
XV.	Field duplicates	<u>N</u>	
XVI.	Field blanks	<u>N</u>	

Note: A = Acceptable  
N = Not provided/applicable  
SW = See worksheet

ND = No compounds detected  
R = Rinsate  
FB = Field blank

D = Duplicate  
TB = Trip blank  
EB = Equipment blank

Validated Samples: \*\* Indicates sample underwent Cat B review.

SOIL

1	LT-C-028-0-2 **	11	MB 480-167179	21		31	
2	LT-C-028-4-6	12		22		32	
3	LT-C-028-8-10	13		23		33	
4	LT-C-029-0-2	14		24		34	
5	LT-C-029-2-4	15		25		35	
6	LT-C-029-8-10	16		26		36	
7	LT-G-001-0-2	17		27		37	
8	LT-G-001-4-6	18		28		38	
9	LT-G-001-10-12	19		29		39	
10		20		30		40	

Notes: \_\_\_\_\_

LDC #: 31414P3b

## VALIDATION FINDINGS WORKSHEET

### Continuing Calibration

Page: 1 of 1

Reviewer: F

2nd Reviewer: 9

METHOD: GC HPLC

Please see qualifications below for all questions answered "N". Not applicable questions are identified as "N/A".

What type of continuing calibration calculation was performed? \_\_\_%D or \_\_\_%R

Y/N N/A Were continuing calibration standards analyzed at the required frequencies?

Y N N/A Did the continuing calibration standards meet the %D / %R validation criteria of  $\leq 20.0\%$  / 80-120%?

**Level IV Only**

Y N N/A Were the retention times for all calibrated compounds within their respective acceptance windows?

[illegible]



LDC #: 313414P3b

# **VALIDATION FINDINGS WORKSHEET** **Initial Calibration Calculation Verification**

Page: 1 of 1  
 Reviewer: F2  
 2nd Reviewer: 9

METHOD: GC ✓ HPLC       

The calibration factors (CF) and relative standard deviation (%RSD) were recalculated using the following calculations:

CF = A/C

Average CF = sum of the CF/number of standards

%RSD = 100 \* (S/X)

Where: A = Area of compound

C = Concentration of compound

S = Standard deviation of calibration factors

X = Mean of calibration factors

#	Standard ID	Calibration Date	Compound	Reported	Recalculated	Reported	Recalculated	Reported	Recalculated
				CF (0.2 std)	CF (0.2 std)	CF (initial)	CF (initial)	%RSD	%RSD
1	KAL-12	12/26/13	PCB 260-1 (2B 35)	298335	298335	249410.929	249410.929	15.0	15.0
			(2B 5)	NR	335020	294840.3	NR	17.9	17.9
				335020		294983.071	294983.1		
2									
3									
4									

Comments: Refer to Initial Calibration findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

LDC #: 31414P3b

# **VALIDATION FINDINGS WORKSHEET** **Continuing Calibration Results Verification**

Page: 1 of 1Reviewer: FJ2nd Reviewer: OLMETHOD: GC ✓ HPLC       

The percent difference (%D) of the initial calibration average Calibration Factors (CF) and the continuing calibration CF were recalculated for the compounds identified below using the following calculation:

$$\% \text{ Difference} = 100 * (\text{ave. CF} - \text{CF}) / \text{ave. CF}$$

Where: ave. CF = initial calibration average CF

CF = continuing calibration CF

A = Area of compound

C = Concentration of compound

#	Standard ID	Calibration Date	Compound	Average CF(Ical)/ CCV Conc.	Reported	Recalculated	Reported	Recalculated
					CF/ Conc. CCV	CF/ Conc. CCV	%D	%D
1	CCV-12 6:16	2/24/14	PCB 260-1 ZB 35	0.5	0.580	0.580	16.0	16.0
			↓ ZB 5	↓	0.5930	0.5930	<del>18.6</del>	18.6
							n	
2								
3								
4								

Comments: Refer to Continuing Calibration findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

LDC #: 31414P2bVALIDATION FINDINGS WORKSHEET  
Surrogate Results VerificationPage: 1 of 1Reviewer: EL2nd reviewer: ELMETHOD: GC HPLC

The percent recoveries (%R) of surrogates were recalculated for the compounds identified below using the following calculation:

% Recovery:  $SF/SS * 100$ Where: SF = Surrogate Found  
SS = Surrogate SpikedSample ID: #1

Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	
TCMY	2P35	0.02	0.0274	137	137	0
DEB	↓	↓	0.0289	144	144	0

Sample ID: \_\_\_\_\_

Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	

Sample ID: \_\_\_\_\_

Surrogate	Column/Detector	Surrogate Spiked	Surrogate Found	Percent Recovery	Percent Recovery	Percent Difference
				Reported	Recalculated	

LDC #: 31414036

## VALIDATION FINDINGS WORKSHEET

Page: 1 of 1Laboratory Control Sample/Laboratory Control Sample Duplicates Results VerificationReviewer: F22nd Reviewer: OLMETHOD: GC HPLC

The percent recoveries (%R) and relative percent differences (RPD) of the laboratory control sample and laboratory control sample duplicate were recalculated for the compounds identified below using the following calculation:

%Recovery = 100 \* (SSC/SA)

RPD = (((SSCLCS - SSCLCSD) \* 2) / (SSCLCS + SSCLCSD)) \* 100

Where

SSC = Spiked sample concentration

LCS = Laboratory Control Sample

SA = Spike added

LCSD = Laboratory Control Sample duplicate

LCS/LCSD samples: LCS 480-167179

Compound	Spike Added (mg/kg)		Spike Sample Concentration (mg/kg)		LCS		LCSD		LCS/LCSD	
					Percent Recovery		Percent Recovery		RPD	
	LCS	LCSD	LCS	LCSD	Reported	Recalc.	Reported	Recalc.	Reported	Recalc.
Gasoline (8015)										
Diesel (8015)										
Benzene (8021B)										
Methane (RSK-175)										
2,4-D (8151)										
Dinoseb (8151)										
Naphthalene (8310)										
Anthracene (8310)										
HMX (8330)										
2,4,6-Trinitrotoluene (8330)										
PCB 1260	2.09	NA	3.14	NA	150	150	NA			

Comments: Refer to Laboratory Control Sample/Laboratory Control Sample Duplicate findings worksheet for list of qualifications and associated samples when reported results do not agree within 10.0% of the recalculated results.

LDC #: 31414036

# VALIDATION FINDINGS WORKSHEET Sample Calculation Verification

Page: 1 of 1  
Reviewer: F7  
2nd Reviewer: al

METHOD: ☒ GC ☐ HPLC

Y N N/A  
Y N N/A

Were all reported results recalculated and verified for all level IV samples?

Were all recalculated results for detected target compounds within 10% of the reported results?

Concentration =  $\frac{(A)(Fv)(Df)}{(RF)(Vs \text{ or } Ws)(\%S/100)}$

Example:

Sample ID. LCS 480-16779 Compound Name 1260

A= Area or height of the compound to be measured  
Fv= Final Volume of extract  
Df= Dilution Factor  
RF= Average response factor of the compound  
In the initial calibration  
Vs= Initial volume of the sample  
Ws= Initial weight of the sample  
%S= Percent Solid

Concentration =  $\frac{0.7509 (10)}{(2.39)}$

= 3.14 mg/kg

#	Sample ID	Compound	Reported Concentrations ( )	Recalculated Results Concentrations ( )	Qualifications
	1260-1 = 173962 = 0.6975		1260-1 = 0.6975		
	249410.929		2 = 0.7332		
			3 = 0.7573		
			4 = 0.8214		
			Ave = 0.7509		

Comments: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

Yes NO N/A

PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER: 31414D SDG# 480 - 53877-2  
LAB: Test America SITE: Glen Island

1.0 Data Completeness and Deliverables

1.1 Has all the data been submitted in CLP deliverable format? 1 /      

1.2 Have any missing deliverables been received and added to the data package? 1 /      

ACTION: Call lab for explanation/resubmittal of any missing deliverables. If lab cannot provide them, note the effect on review of the data in the reviewer narrative.

2.0 Cover Letter, SDG Narrative

2.1 Is a laboratory narrative or cover letter present? 1 /      

2.2 Are the case number and/or SDG number contained in the narrative or cover letter? 1 /      

3.0 Data Validation Checklist

3.1 Does this data package contain:

Water data?    /   

Waste data?    /   

Soil/solid data? /      

POLYCHLORINATED BIPHENYLS

1.0 Traffic Reports and Laboratory Narrative

1.1 Are traffic report and chain-of-custody forms present for all samples? 1 /

Yes NO N/A

ACTION: If no, contact lab for replacement of missing or illegible copies.

- 1.2 Do the traffic reports, chain-of-custody forms or SDG narrative indicate any problems with sample receipt, condition of the samples, analytical problems or special circumstances affecting the quality of the data?

✓ [ ]

ACTION: If any sample analyzed as a soil, other than TCLP, contains 50%-90% water, all data should be qualified as estimated, "J." If a soil sample, other than TCLP, contains more than 90% water, non detects shall be qualified as unusable, "R."

ACTION: If samples were not iced or if the ice was melted upon arrival at the laboratory and the temperature of the cooler was elevated (> 10° C), flag all positive results "J" and all non-detects "UJ".

## 2.0 Holding Times

- 2.1 Have any PCB technical holding times, determined from date of collection to date of extraction, been exceeded?

✓ [ ]

Water and waste samples for PCB analysis must be extracted within 7 days of the date of collection. Extracts must be analyzed within 40 days of the date of extraction. Soils and solid samples must be extracted within 14 days of collection and analyzed within 40 days of extraction.

ACTION: If technical holding times are exceeded, flag all positive results as estimated, "J," and sample quantitation limits "UJ" and document in the narrative that holding times were exceeded. If analyses were done more than 14 days beyond holding time, either on the first analysis or upon re-analysis, the reviewer must use professional judgement to determine the reliability of the data and the effects of additional storage on the sample results. At a minimum, all the data should at least be

Yes NO N/A

qualified "J", but the reviewer may determine that non-detects are unusable, "R." (Table 1)

Table 1. Holding Time Criteria

Matrix	Preserved	Criteria	Action	
			Detected compounds	Non-detected compounds
Aqueous	No	$\leq 7$ days (extraction) $\leq 40$ days (analysis)	J*	UJ*
	No	$> 7$ days (extraction) $> 40$ days (analysis)	J	UJ
	Yes	$\leq 7$ days (extraction) $\leq 40$ days (analysis)	No qualification	
	Yes	$> 7$ days (extraction) $> 40$ days (analysis)	J	UJ
	Yes/No	$> 28$ days (gross exceedance)	J	R
Non-aqueous	No	$\leq 14$ days (extraction) $\leq 40$ days (analysis)	J*	UJ*
	No	$> 14$ days (extraction) $> 40$ days (analysis)	J	UJ
	Yes	$\leq 14$ days (extraction) $\leq 40$ days (analysis)	No qualification	
	Yes	$> 14$ days (extraction) $> 40$ days (analysis)	J	UJ
	Yes/No	$> 28$ days (gross exceedance)	J	R

\* only if cooler temperature exceeds 10°C; no action required if cooler temperature < 10°C.

### 3.0 Surrogate Recovery (Form II/Equivalent)

3.1 Were the recoveries of tetrachloro-m-xylene (TCMX) and decachlorobiphenyl (DCB) presented on CLP Surrogate Recovery Summary forms (Form II), or equivalent, for each of the following matrices?

a. Water/Waste

11 — /



Yes ☒ NO ☐ N/A ☐

b. Soil/Solid

3.2 Are all the PCB samples listed on the appropriate surrogate recovery form for each of the following matrices?

a. Water

☒ ☐ ☐

b. Waste

☒ ☐ ☐

c. Soil/Solid

☒ ☐ ☐

ACTION: Call lab for explanation/resubmittals.  
If missing deliverables are unavailable,  
document the effect in the data assessment.

3.3 Are all recovery limits for the surrogates TCMX and DCB between 30-150% for all samples, including MS and MSDs, LCSS and all blanks?

☒ ☐ ☐

Note: Reviewer shall use lab in-house recovery limits, if available. In-house criteria should be examined for reasonableness.

ACTION: Circle all outliers in red. Follow surrogate criteria, Table 2.

Note: DCB is used when PCBs are determined as Aroclors. DCB is the internal standard when determining PCB congeners and TCMX the surrogate.

3.4 Were surrogate retention times (RT) within the windows established during the initial 5-point analysis?

☒ ☐ ☐

ACTION: Follow surrogate criteria, Table 2.

Table 2. Surrogate Recovery Criteria

Criteria	Action	
	Detected Target Compounds	Non-detected Target Compounds
%R > 200%	J	Use professional judgement

Yes NO N/A

150% < %R ≤ 200%	J	No qualification
30% ≤ %R ≤ 150%	No qualification	
10% ≤ %R < 30%	J	UJ
%R < 10% (sample dilution not a factor)	J	R
%R < 10% (sample dilution is a factor)	Use professional judgement	
RT out of RT window	Use professional judgement	
RT within RT window	No qualification	

3.6 Are there any transcription/calculation errors between raw data and Form II?

1 1 ✓   

ACTION: If large errors exist, call lab for explanation/resubmittal. Make any necessary corrections and document the effect in data assessments.

#### 4.0 Laboratory Control Sample (LCS)

4.1 Are raw data and percent recoveries present for all Laboratory Control samples as required by Method 8000B (section 8.5) and Method 8082A (section 8.4.2)?

1 ✓      

Verify that QC check samples were extracted and analyzed by the same procedures used for the actual samples.

ACTION: If any Laboratory Control Sample data are missing, call the lab for explanation/resubmittals. Make note in the data assessment.

NOTE: For aqueous samples, an additional QC check sample must be prepared and analyzed when any analyte in a matrix spike fails the required acceptance criteria (see section 5.3 below).

Yes NO N/A

The additional QC check sample must contain each analyte that failed in the MS analysis.

Note: When the results for matrix spike analysis indicates a problem due to sample matrix effects, the LCS results are used to verify the laboratory can perform the analysis in a clean sample.

4.2 Were Laboratory Control Samples analyzed at the required concentration as specified in Method 8000B(sec 8.5) for all analytes as specified in Table 3.

☒ 1 \_ \_

Note: Use lab in-house criteria, if available.

ACTION: If Laboratory Control Samples were not analyzed at the required concentration or the required frequency, make note in the data assessment and use professional judgement to determined the affect on the data.

4.3 Were the LCS recoveries within the percent recoveries as specified in Table 3.

☒ 1 \_ \_

Table 3. LCS Criteria

Compound	% Recovery
Aroclor 1016	50-150
Aroclor 1260	50-150
Tetrachloro-m-xylene (surrogate)	30-150
decachlorobiphenyl (surrogate)	30-150

4.4 If no, were Laboratory Control Samples re-analyzed?

☐ 1 \_ ☒

ACTION: If QC check samples were not re-analyzed, or a general system problem is indicated by repeated failure to meet the QC acceptance criteria specified in the method, make note in the data assessment and use Table 4 recovery actions criteria.

#### Table 4. LCS Recovery Actions

Criteria	Action	
	Detected Associated Compounds	Non-Detected Compounds
%R > Upper Acceptance Limit	J	No qualification
%R < Lower Acceptance Limit	J	R
Lower Acceptance Limit $\leq$ %R $\leq$ Upper Acceptance Limit	No qualifications	

5.1 Are all data for one matrix spike and matrix duplicate (unspiked) pair (MS/Dup) or matrix spike/matrix duplicate (MS/MSD) present and complete for each matrix (Method 8082A Section 8.4.1)?   

5.2 Have MS/Dup or MS/MSD results been summarized on modified CLP Form III?

5.3 Were matrix spikes analyzed at the required frequency for each of the following matrices? (One MS/Dup, MS/MSD must be performed for every 20 samples of similar matrix or concentration level. Laboratories analyzing one to ten samples per month are required to analyze at least one MS per month (Method 8000B-39 (section 8.5)).

a. Water

b. Waste

Yes NO N/A  
☐ ☐ ☒

c. Soil/Solid

☐ ☐ ☐

ACTION: If any MS/Dup or MS/MSD data are missing,  
take the action specified in 3.2 above.

- 5.4 Were Laboratory Control Samples analyzed  
for all analytes as specified in Table 5,  
or did the lab use the optional QC acceptance  
criteria i.e., in-house criteria?

☒ ☐ ☐

List the criteria used and make note in  
data assessment.

Criteria used \_\_\_\_\_

Table 5. MS/MSD Criteria

Compound	Percent Recovery QC Limits	RPD
Aroclor 1016	29-135	0-15
Aroclor 1260	29-135	0-20

- 5.5 Was the matrix spike prepared at the proper spike  
concentration? (Method 8000B, section 8.5.1-8.5.2)

☒ ☐ ☐

For aqueous organic extractable, the spike concentration  
should be prepared according options in: Method 8000B-40,  
(section 8.5.1 and 8.5.2).

- 5.6 Were the matrix spike and matrix spike duplicate recovery and RPD  
limits met as specified in Table 5. Note: No qualification of the  
data is necessary on MS and MSD data alone. Use professional  
judgement to use the MS and MSD results in conjunction with other  
QC criteria to determine the need for some qualification of the  
data. If any MS and MSD, percent recovery, or RPD results in the  
Aroclor fraction is out of specification (Table 5), qualify data  
to include the consideration of the existence interference in the  
raw data. In some instances it may be determined that only the  
replicate or spiked samples are affected. Alternatively, the  
data may suggest that the laboratory is having a systematic  
problem with one or more analytes, thereby affecting all  
associated samples. Use professional judgement to determine the  
need for qualifications of detects of non-spiked compounds.

Yes NO N/A

☒ ☐ ☐

Table 6. MS/MSD Actions for Analysis

Criteria	Action	
	Detected Associated Compounds	Non-Detected Compounds
%R or RPD > Upper Acceptance Limit	J	No qualification
20% ≤ %R < Lower Acceptance Limit	J	UJ
%R < 20%	J	Use professional judgement
Lower Acceptance Limit ≤ %R ≤ Upper Acceptance Limit	No qualifications	

6.0 Blanks (Form IV/Equivalent)

6.1 Was reagent blank data reported on CLP equivalent Method Blank Summary form(s) (Form IV)?

☒ ☐ ☐

6.2 Frequency of Analysis: Has a reagent blank been analyzed for every 20 (or less) samples of similar matrix or concentration or each extraction batch?

☒ ☐ ☐

Note: Method blank should be analyzed, either after the calibration standard or at any time during the analytical shift.

ACTION: If any blank data are missing, take action as specified above (section 3.2) . If blank data is not available, reject (R) all associated positive data. However, using professional judgement, the data reviewer may substitute field blank data for missing method blank data.

6.3 Chromatography: review the blank raw data - chromatograms, quant reports or data system

Yes NO N/A

printouts.

Is the chromatographic performance (baseline stability) for each instrument acceptable for PCBs?

☒ ☐ ☐

#### 7.0 Contamination

NOTE: "Water blanks", "distilled water blanks" and "drilling water blanks" are validated like any other sample and are not used to qualify the data. Do not confuse them with the other QC blanks discussed below.

7.1 Do any method/instrument/reagent/cleanup blanks have positive results for PCBs? When applied as described below, the contaminant concentration in these blanks are multiplied by the sample Dilution Factor and corrected for % moisture when necessary.

☐ ☒ ☐

7.2 Do any field/rinse blanks have positive PCB results?

☐ ☐ ☒

ACTION: Prepare a list of the samples associated with each of the contaminated blanks. (Attach a separate sheet.)

NOTE: All field blank results associated to a particular group of samples (may exceed one per case or one per day) may be used to qualify data. Blanks may not be qualified because of contamination in another blank. Field blanks must be qualified for surrogate, or calibration QC problems.

ACTION: Follow the directions in Table 7 below to qualify sample results due to contamination. Use the largest value from all the associated blanks.

Table 7. Blank Contamination Criteria

Blank Type	Blank Result	Sample Result	Action for Samples
------------	--------------	---------------	--------------------

Yes NO N/A

Method, Clean up, Instrument, Field	Detects	Not detected	No qualification
	< CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL	No qualification
	> CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL and < blank contamination	Report the concentration for the sample with a U
		≥ CRQL and ≥ blank contamination	No qualification
	= CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL	No qualification
	Gross contamination	Detects	Qualify results as unusable R

Note: Analytes qualified "U" for blank contamination are treated as "hits" when qualifying for calibration criteria.

Note: When applied as described in Table 7 above, the contaminant concentration in the blank is multiplied by the sample dilution factor.

NOTE: If gross blank contamination exists(e.g., saturated peaks, "hump-o-grams," "junk" peaks), all affected positive compounds in the associated samples should be qualified as unusable "R", due to interference. Non-detected pesticide target compounds do not require qualification unless the contamination is so high that it interferes with the analyses of non-detected compounds.

7.3 Are there field/rinse/equipment blanks associated with every sample?

11 ✓

ACTION: For low level samples, note in data assessment that there is no associated field/rinse/equipment blank. Exception: samples taken from a drinking water tap do not have associated field blanks.



Yes NO N/A

8.0 Gas Chromatography with Electron Capture Detector (GC/ECD) Instrument Performance Check (CLP Form VI and Form VII Equivalent)

8.1 Was the proper gas chromatographic capillary column used for the analysis of PCBs? ☒ 1

Action: Check raw data, instrument logs, or contact the lab to determine what type of columns were used. (Method 8082, section 4.2)

8.2 Indicate the specific type of narrow bore or wide bore (.53 mm ID, fused silica GC columns, such as DB-608 and DB-1701 or equivalent).

column 1: DB-5

column 2: DB-35

ACTION: Note any changes to the suggested materials in section 8.1 above in the data assessment. Also note the impact (positive or negative) such changes have on the analytical results.

9.0 Calibration and GC Performance

9.1 Are the following Gas Chromatograms and Data Systems Printouts for both columns present for all samples, blanks, MS, replicates?

- |  |                                       |     |                                     |
|--|---------------------------------------|-----|-------------------------------------|
| a. Samples                             | <input checked="" type="checkbox"/> 1 | ___ | ___                                 |
| b. All blanks                          | <input checked="" type="checkbox"/> 1 | ___ | ___                                 |
| c. Matrix spike samples                | <input type="checkbox"/> 1            | ___ | <input checked="" type="checkbox"/> |
| d. 5 pt. initial calibration standards | <input checked="" type="checkbox"/> 1 | ___ | ___                                 |
| e. calibration verification standards  | <input checked="" type="checkbox"/> 1 | ___ | ___                                 |
| f. Laboratory Control samples (LCS)    | <input checked="" type="checkbox"/> 1 | ___ | ___                                 |

ACTION: If no, take action specified in 3.2 above.

9.2 Are data summary forms (containing calibration factors or response factors) for the initial 5

Yes NO N/A

pt. calibration and daily calibration verification  
standards present and complete for each column  
and each analytical sequence?

1,1 / — —

Note: Calibration Aroclor mixtures other than 1016/1260  
may be used (as per approved project QA plan)

NOTE: If internal standard calibration procedure is  
used (Method 8000B-15 (section 7.4.2.2)), then  
response factors must be used for %RSD  
calculations and compound quantitation. If,  
external standard calibration procedures are  
used (Method 8000B-16 (section 7.4.2.1)),  
then calibration factors must be used. The  
internal standard approach is highly  
recommended for PCB congener analysis.

ACTION: If any data are missing or it cannot be  
determined how the laboratory calculated  
calibration factors or response factors,  
contact the lab for explanation/resubmittals.  
Make necessary corrections and note any  
problems in the data assessment.

9.3 Are there any transcription/calculation errors  
between raw data and data summary forms?

— 1,1 / —

ACTION: If large errors exist, call lab for  
explanation/resubmittal, make necessary  
corrections and document the effect in data  
assessments.

9.4 Are standard retention time (RT) windows for each  
PCB peak of interest presented on modified CLP  
summary forms?

1 / — —

ACTION: If any data are missing, or it cannot be  
determined how RT windows were calculated,  
call the lab for explanation/resubmittals.  
Note any problems in the data assessment.

NOTE: Retention time windows for all PCBs are  
established using retention times from three  
calibration standards analyzed during the  
entire analytical sequence (Method 8000B,  
section 7.6). Best results are obtained

Yes NO N/A

using retention times which span the entire sequence; i.e., using the calibration verification/continuing calibration standards analyzed every 12 hours.

- 9.5 Were RT windows on the confirmation column established using three standards as described above?

☒ 1 \_ \_

NOTE: RT windows for the confirmation column should be established using a 3 pt. calibration, preferably spanning the entire analytical sequence as described in 9.4 above. If RT windows on one column are tighter than the other, this may result in false negatives when attempting to identify compounds in the samples.

ACTION: Note potential problems, if any, in the data assessment.

- 9.6 Do all standard retention times in each level of the initial 5 pt. calibrations for PCBs fall within the windows established during the initial calibration sequence?

☒ 1 \_ \_

ACTION i: If no, all samples in the entire analytical sequence are potentially affected. Check to see if three standard spanning the entire sequence were used to obtained RT windows. If the lab used three standards from the 5 pt., RT windows may be too tight. If so, RT windows should be recalculated as per Method 8081B-15 (section 7.4.6).

- ii. Alternatively, check to see if the chromatograms contain peaks within an expanded window surrounding the expected retention times.

If no peaks are found and the surrogates are visible, non-detects are valid. If peaks are present but cannot be discerned through pattern recognition or by using revised RT windows, qualify all positive results and non-detects as unusable, "R".

- 9.7 Has the linearity criteria for the initial calibration standards been satisfied for both

columns? (% RSD for the calibration factors (CFs)  
for the three to five major peaks of each of the  
Aroclor compounds must be < 20.0%).

Yes NO N/A

☒ 1 ☐ ☐

ACTION: If no, follow Table 8 criteria.

Table 8. Initial Calibration CF Action for Aroclor Analysis

Criteria	Action	
	Detected Associated Compounds	Non-Detected Associated Compounds
% RSD > 20%	J	UJ
% RSD within allowable limits	No qualifications	

9.8 Does the calibration verification/continuing  
calibration standard contain the PCB peaks of  
interest, analyzed on each working day, prior  
to sample analyses (Method 8082, sections 7.6.2)? ☒ 1 ☐

9.9 Has a calibration verification/continuing calibration  
standard been analyzed after every 10 samples and at  
the end of each analytical sequence  
(Method 8082A, section 7.6.2). ☒ 1 ☐

ACTION: If no, take action as specified in section  
3.2 above.

9.10 Has the percent difference (%D) between the  
Calibration Factor (CF) of each of the three to  
five peaks used to identify the Aroclor in the  
CCV and the CF from these peaks in the initial  
calibration exceeded  $\pm 15\%$  ~~20%~~ ☒ 1 ☐

9.11 Has a new 5 pt. initial calibration curve been generated  
for those PCB analytes which failed in the calibration  
verification/continuing calibration standard (8000B, section  
7.7.3), and all samples which followed the out-of-control

calibration verification/standard continuing calibration  
Standard? Yes NO N/A  
[1] ☒ ☐

ACTION: If the %D for any analyte exceeded the  $\pm 15\%$  <sup>20%</sup> criterion and the instrument was not recalibrated for those analytes, qualify positive results for all associated samples (those which followed the out-of-control standard) "J" and sample quantitation limits "UJ". (see Table 9)

9.12 Have retention time (RT) windows been properly calculated for each analyte of interest (Method 8000B, section 7.6), using RTs from the associated calibration verification/continuing standard? ☒ ☐

ACTION: If no, take action specified in section 3.2 above

9.13 Do all standard retention times for each calibration verification/continuing calibration standard fall within the windows established during the initial calibration sequence? ☒ ☐

9.14 Do all standard retention times for each mid-concentration standard (analyzed after every 10 samples) fall within the daily RT windows. ☒ ☐

ACTION: For any multi-response analytes, retention time windows should be used but analyst and reviewer should rely primarily on pattern recognition or use paragraph B below. If the answer to either 9.13 or 9.14 above is no, check the chromatograms of all samples which followed the last in-control standard. If samples were not re-analyzed, all samples analyzed after the last in-control standard must be evaluated using professional judgement.

(A) For non-detected target compounds, check to see if the sample chromatograms contain any peaks that are close to the expected RT window of the Arcolor of interest. If no peaks are present, no qualification of data is necessary. If peaks are present close th RT window of the Aroclor of interest, qualify the non-detected values as presumptively present "N".

Yes NO N/A

- (B) For detected compounds in the affected samples, if peaks within the RT window, no qualification necessary. If peaks are close to the expected RT window of the Aroclor of interest, the reviewer can examine the data package for the presence of three or more standards the Aroclor of interest that were run within the analytical sequence during which the sample was analyzed. If three or more such standards are present, the RT window can be reevaluated using the Mean Retention Times of the standards. If the peaks in the affected sample fall within the revised window, qualify the detected target compounds "NJ". If the reviewer cannot do anything with the data to resolve the problem of concern, qualify all non-detects as unusable "R". (Table 9)

- 9.15 Has no more than 12 hours elapsed from the injection of the opening CCV and the end of the analytical sequence sequence (closing CCV). (Table 9) 11 ☒

Table 9. CCV Criteria

Criteria	Action	
	Detected Associated Compounds	Non-Detected Associated Compounds
RT out of RT window	Use professional judgement (Sec 9.14)	
%D not within +/- 15%	J	UJ
Time elapsed greater than section 9.15 criteria.	R	
%D, time elapsed, RT are all within acceptable limits.	No qualifications	

- 9.16 Are there any transcription/calculation errors between raw data and data summary forms?      ☒

ACTION: If large errors exists, call lab for explanation/resubmittal, make any necessary corrections and document the effect in data assessments under "Conclusions".

10.0 Analytical Sequence Check (Form VIII-PEST/Equivalent)

- 10.1 Have all samples been listed on CLP Form VIII or equivalent, and are separate forms present for each column? 11 ☒

Yes NO N/A

ACTION: If no, take action specified in 3.2 above.

10.2 Was the proper analytical sequence followed for each initial calibration and subsequent analyses?

☒ 1 — —

ACTION: If no, use professional judgement to determine the severity of the effect on the data and qualify it accordingly. Generally, the effect is negligible unless the sequence was grossly altered or the calibration was also out of limits.

10.3 Were the TCMX/DCB surrogate RTs for the samples within the mean surrogate RT from the initial calibration?

☒ 1 — —

Action: If no, see "Action" in section 9.14 above

11.0 Extraction Techniques for Sample Preparation

Method 8082A permits a variety of extraction techniques to be used for sample preparation. Check which extraction procedure was used?

1. Aqueous samples: *N/A*

- |  |   |
|--|---|
| 1. Separatory funnel (Method 3510)                   | <input type="checkbox"/> — —            |
| 2. Continuous liquid-liquid extraction (Method 3520) | <input type="checkbox"/> — —            |
| 3. Solid phase extraction (Method 3535)              | <input checked="" type="checkbox"/> — — |
| 4. Other   | <input type="checkbox"/> — —            |

2. Solid samples:

- |  |   |
|--|---|
| 1. Soxhlet (Method 3540)               | <input type="checkbox"/> — —            |
| 2. Automated Soxhlet (Method 3541)     | <input type="checkbox"/> — —            |
| 3. Pressurized fluid (Method 3545)     | <input type="checkbox"/> — —            |
| 4. Microwave extraction (Method 3546)  | <input type="checkbox"/> — —            |
| 5. Ultrasonic extraction (Method 3550) | <input checked="" type="checkbox"/> — — |

Yes NO N/A

6. Supercritical fluid (Method 3562) ☐ ☐ ☐

7. Other ☐ ☐ ☐

11.1 Extract Cleanup - Efficiency Verification (Form IX/Equivalent)

11.1.1 Method 8082 (section 7.2) references method 3660 (sulfur) and 3665A (sulfuric acid) to use for cleaning extracts. Were one or both method used? ☒ ☐ ☐

ACTION: If no, take action specified in 3.2 above.  
If data suggests cleanup was not performed, make note in the data assessment.

NOTE: Method 3620A, Florisil, may be used per approved project QA plan. The method does not list which analytes and surrogate(s) to use to verify column efficiency. The reviewer must check project plan to verify method used as well as the correct PCB list. If not stated or available, use the CLP listing or accept what the laboratory used.

11.2 Are all samples listed on modified CLP PCBs Florisil/Cartridge Check Form? ☐ ☐ ☒

ACTION: If no, take action specified in 3.2 above.

11.3 Was GPC Cleanup (method 3640A) performed? ☐ ☐ ☒

NOTE: GPC cleanup is not required and is optional. The reviewer should check Project Plan to verify requirement.

11.4 Were the same PCB analytes used in calibration used to check the efficiency of the cleanup procedures? ☐ ☐ ☒

11.5 Are percent recoveries (% R) of the PCBs and surrogate compounds used to check the efficiency of the cleanup procedures within lab's in-house QC limits (use 70-130% if not available). ☐ ☐ ☒



Yes NO N/A

70-130% for GPC calibration?

☐ ☐ ☒

Qualify only the analyte(s) which fail the recovery criteria as follows:

ACTION: If % R are < 70%, qualify positive results "J" and quantitation limits "UJ". Non-detects should be qualified "R" if zero %R was obtained for PCBs. Use professional judgement to qualify positive results if recoveries are greater than the upper limit.

#### 12.0 PCB Identification

12.1 Has CLP Form X or equivalent, showing retention time data for positive results on the two GC columns, been completed for every sample in which a PCB was detected?

☒ ☐ ☐

ACTION: If no, take action specified in 3.2 above, or compile a list comparing the retention times for all sample hits on the two columns.

12.2 Are there any transcription/calculation errors between raw data and data summary forms (initial calibration summaries, calibration verification summaries, analytical sequence summaries, GPC and cleanup verification forms)?

☐ ☒ ☐

ACTION: If large errors exist, call lab for explanation/resubmittal, make necessary corrections and note error in the data assessment.

12.3 Are retention times (RT) of sample compounds within the established RT windows for both columns/analyses?

☒ ☐ ☐

ACTION: Qualify as unusable (R) all positive results which were not confirmed by second GC column analysis. Also qualify "R", unusable, all positive results not within RT windows unless associated standard compounds are similarly biased. The reviewer should use professional judgement to assign an appropriate quantitation limit.

Yes NO N/A

12.4 Check chromatograms for false negatives, especially if RT windows on each column were established differently.

Were there any false negatives?

11 ☒ 1

ACTION: Use professional judgement to decide if the compound should be reported. If there is reason to believe that peaks outside retention RT windows should be reported, make corrections to data summary forms (Form I) and note in data assessment.

12.5 Was GC/MS confirmation provided when sample concentration was sufficient (> 10 ug/ml) in the final extract?

11 ☒ 1

ACTION: Indicate with red pencil which Form I results were confirmed by GC/MS and also note in data assessment. GC/MS confirmation is an option, see section 7.10 of Method 8082A-20. If GC/MS confirmation is not available, follow action in section 3.2.

12.6 Is the percent difference (%D) calculated for the positive sample results on the two GC columns <25.0%?

11 ☒ 1  
All MD

NOTE: The method requires quantitation from one column. The second column is to confirm the presence of an analyte. It is the reviewer's responsibility to verify from the project plan what the lab was required to report. If the lab was required to report concentrations from both columns, continue with validation for % Difference. If required, but not reported, either contact the lab for results or calculate the concentrations from the calibration. If not required, skip this section. Document actions in Data Assessment.

ACTION: If the reviewer finds neither column shows interference for the positive hits, the data should be qualified as follows:

% Difference

Qualifier

Yes NO N/A

0-25%	none
26-70%	"J"
71-100%	"NJ"
101-200% (No Interference)	"R"
101-200% (Interference detected)	"NJ"
>50% (PCBs value is <CRQL)	"U"
>200%	"R"

Note: The lower of the two values is reported on Form I. If using professional judgement, the reviewer determines that the higher result was more acceptable, the reviewer should replace the value and indicate the reason for the change in the data assessment.

### 13.0 Compound Quantitation and Reported Detection Limits

13.1 Are there any transcription/calculation errors in Form I results? Check at least two positive values. Were any errors found?   1  

NOTE: Single-peak PCBs results can be checked for rough agreement between quantitative results obtained on the two GC columns. The reviewer should use professional judgement to decide whether a much larger concentration obtained on one column versus the other indicates the presence of an interfering compound. If an interference is suspected, the lower of the two values should be reported and qualified according to section 12.6 above. This necessitates a determination of an estimated concentration on the confirmation column. The narrative should indicate that the presence of interferences has led to the quantitation of the second column confirmation results.

13.2 Are the EDLs (Estimated Detection Limits) adjusted to reflect sample dilutions and, for soils, % moisture?   1  

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and document effect in data assessments.

Yes NO N/A

ACTION: When a sample is analyzed at more than one dilution, the lowest EDLs are used (unless a QC exceedance dictates the use of the higher EDL data from the diluted sample analysis). Replace concentrations that exceed the calibration range in the original analysis by crossing out the value on the original Form I and substituting it with data from the analysis of diluted sample. Specify which Form I is to be used, then draw a red "X" across the entire page of all Form I's that should not be used, including any in the summary package.

ACTION: EDLs affected by large, off-scale peaks should be qualified as unusable, "R". If the interference is on-scale, the reviewer can provide a modified EDL flagged "UJ" for each affected compound.

14.0 Chromatogram Quality

14.1 Were baselines stable?

☒ 1 — —

14.2 Were any electropositive displacement (negative peaks) or unusual peaks seen?

— ☒ 1 —

ACTION: Note all system performance problems in the data assessment.

15.0 Field Duplicates

15.1 Were any field duplicates submitted for PCB analysis?

☐ 1 ☒ 1 —

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between field duplicate results must be addressed in the reviewer narrative. However, if large differences exist, the identity of the field duplicates is questionable. An attempt should be made

Yes NO N/A

to determine the proper identification of  
field duplicates.